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PROVISIONAL SPECIFICATION.

DUPLICATE

Improvements relating to the Treatment of Leaves, Stems, and other Parts of Plants for Extracting and Utilizing the Aromatic Principles of the same, especially in the Manufacture of Alcoholic Liquors.

I, GEORGE EUGÈNE JACQUEMIN, of Malzéville, near Nancy, in the Republic of France, Agricultural Chemist, do hereby declare the nature of this invention to be as follows:—

The leaves of plants at various epochs of vegetable life are the seat of formation of certain principles which the plant utilizes for the benefit of some of its other organs, either immediately, or after having kept them in reserve for a time with the object of yielding them up at the opportune moment. To instance one fact, fruit is apt to acquire a characteristic flavour at the time of maturation, owing, in all probability, to the advent of this principle, the odour characterizing which is noticeable in the black currant leaf, for example; or the aromatic principle may have formed and become split up into two distinct principles, as in the case of apple-trees, pear-trees and the like where the odour or flavour of the fruit is not noticeable in the leaf beforehand.

The leaves of many plants, the fruit of which are clearly distinguishable by a flavour of their own, exhibit nothing whereby the prime cause of this well-defined flavour can be so much as surmised. Neither by crumpling or rolling them between the fingers, nor by pounding them in a porcelain mortar does the slightest odour or flavour of an aromatic or fragrant character become perceptible; and even by boiling them in water the flavour will generally not be rendered any more noticeable.

It has occurred to me that these principles peculiar to certain leaves with regard to which the nature or operation which I am inclined to attribute to them,—as co-existent with the process of maturation of the fruit,—is not at all revealed in the leaf, might be likened to glucosides, as there is reason to suppose that bodies of this description which make their appearance in the fruit, at a certain period of life of the plant, are confronted therein by a diastase, under the influence of which they become split up into glucose or saccharine matter and into an aromatic principle which imparts the peculiar character to the distinguishing flavour of the fruit.

Having explained this much by way of introduction, the object of the invention herein described is a practical application of the principles stated to the requirements of the manufacturing industry.

This I propose to effect by the following process.

I immerse leaves, say, of an apple tree or pear tree into a saccharine liquid of such density as to enable fermentation to commence and to terminate normally. I then add some leaven or saccharomyces so selected as to ensure fermentation but not to bring out the fragrance or bouquet of the fruit. As soon as fermentation has set in there becomes very clearly noticeable an odour

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of apples or pears according to the kind of leaves treated, and by distilling these a species of brandy will be obtained, characterised by a fine flavour of the corresponding fruit

This result clearly shows that the leaven, by means of a diastase, which it no doubt excretes, effects the division of this glucoside *i.e.* this principle peculiar to the leaves, into a special aromatic product and into sugar which will ferment together with the sugar on the liquid acting as a medium in this cellular life. It must however be understood that I claim the ownership of the process herein described no matter what theoretical explanation may be offered to account for the practical results which it permits to be attained.

A fermentation of the same kind obtained with vine leaves will yield a liquor of a vinous odour, and, by distillation, a finely flavoured brandy. It will be understood that its flavour will be more delicate, the better the quality or nature of the stock from which the leaves are taken, or the better the wine which would in the ordinary cause have been obtained from such stock.

For all these fermentations of the leaves of diverse fruit bearing plants I have found that the odour of the aromatic principle extracted from the liquor was more marked, or intense, when distillation was proceeded with before fermentation was quite completed. On the other hand, as these aromatic principles are very volatile, a great deal of them is evolved during the fermentation process. Now it is important that the greatest possible quantity of this aroma should be intercepted, and to this end it will be necessary to send the gases produced by fermentation through a condenser charged with alcohol, which will dissolve the aroma thus given off; and the odorous gases should be conducted through the entire apparatus with a view to separating the essential oils and intercepting even the faintest perfume which they carry with them.

It must further be understood that my invention comprises the application of the treatment just described not to leaves only but to all green portions of the plant, from such as stalks shoots sprouts, the tendrils of vine foliage or the like. Also, the method of operation just described is capable of undergoing a modification which I will now indicate.

Instead of submitting the leaves charged with the principles in question to fermentation, the aromatic principles may first be extracted from the leaves by decoction, infusion, digestion or any other treatment, with water, sacchariferous must or any other suitable liquor and then to induce fermentation in the presence of the aromatic principles thus treated and separated from the leaves.

This variant of my process involves no alteration in the general spirit in which it was conceived, seeing that the alcoholic fermentation which takes place, in any case affects only the particular principles contained in the leaves. The operation, therefore, takes the same course irrespective of whether the aromatic principles are still contained in the leaves or have been first separated therefrom.

I shall now state how I prefer to proceed in practice, taking the treatment of vine leaves as an instance.

The leaves or the foliage (including stalks tendrils and the like) are treated with boiling water or boiling saccharine must wherein they are left to infuse for a number of hours. The must is separated from the leaves either before or after the complete cooling of the liquid, and having been brought to such a degree of richness in saccharine matter as will enable fermentation to commence and terminate in a normal manner, the leaven selected for the purpose is added to it, and it is then allowed to ferment.

A good result will also be obtained by boiling the leaves in the liquors above named with or without subsequent infusion, or simply by causing the leaves to infuse or digest in a suitable liquid at a temperature below 100° Centigrade. The effect of these modifications will be simply to vary the degree of delivery of the finer product.

Also, the quantity of vine leaves to be employed for every hectolitre of must to be fermented, will vary according to the result it is desired to obtain. It

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would seem that a proportion of from 2 to 5 kilogrammes of leaves per hectolitre should be recommended where it is proposed subsequently to distil the fermented liquor with a view to obtaining brandy. I may mention that by the expression "saccharine (or sacchariferous) must" I mean must containing sugar
 5 no matter of what origin or from what source, such as beet-sugar, glucose or the like.

All that has been stated above with regard to vine leaves equally applies to the leaves of apple trees pear and other trees.

Instead of fresh leaves, leaves which have been preserved by proper methods
 10 and brought from distant places, may be similarly treated.

Of the methods of preservation of leaves, the following appear to merit the preference:

(1) The leaves are heaped in a cask, barrel or similar vessel, and a suitable proportion,—say about 5 or 10 *per cent.*—of leavening must, in full fermentation is poured over them.
 15

(2) Upon the heap of leaves there is further poured a syrup of sugar or glucose of as high a degree of concentration as possible; or glycerine may be used instead; all the leaves being thoroughly flushed by or submerged in the liquor. The leaves thus treated will for a long time keep in a serviceable condition in
 20 a close vessel; while preservation may still further be assisted by an addition of an organic acid or an antiseptic.

Whatever the mode of preservation adopted, the leaves preserved will have to be used or treated after the same method that has been described with reference to fresh leaves.

25 My process also enables vine leaves, either from the same vine, or from a superior stock, to be used for the purpose of improving ordinary grape juice wines. The method of operation I adopt in this case is as follows:

The vine leaf in which are present,—either in the condition of glucosides or in any other forms,—those aromatic principles which, by migrating into the
 30 grape will presently contribute an important share to the development to the bouquet of the wine, may be employed either directly or indirectly for the amelioration of wines.

I have found that by means of vine leaves from a stock of recognised excellence, added to a somewhat poor vintage, the vinosity or bouquet of the wine
 35 from such vintage may be enhanced, by either leaving the wine to ferment spontaneously or by inducing its fermentation by means of leaven carefully selected from good stock. In the latter case the evolution produced by the leaven will be much more effective than it would be in ordinary wine juice left alone, because the leaf taken from a good stock brings along with it and imparts to
 40 the most certain nutritive elements calculated to assist the evolution of the leaven selected in a marked degree.

There are two ways of turning these observations to account, one consisting in adding from 100 to 500 grammes, or more, of vine leaves, per hectolitre of vintage to be treated; such leaves being either of the same or a superior stock,
 415 and having simply been washed first with acidulated water, and then with pure water, so as to get rid of any dust or foreign elements that may be adhering to them. The leaves will prove more effective if care be taken first to chop them very finely by means of any suitable instrument or converting them into a pulp before mixing them with the grapes of the vintage. Leaves which have
 50 been preserved, no matter by what method, will have the same effect.

Or, preferably, infusions, decoctions or extracts of leaves, prepared beforehand in the following manner, may be used. The leaves after having been thoroughly cleansed, as above alluded to, or undergone any other convenient cleaning or purifying process, are chopped, pounded, or otherwise divided,—or
 55 they may be left undivided,—are placed in an apparatus not unlike what is known as a diffuser, or in any other apparatus permitting their active principles to be extracted by means of cold or hot water acidulated (preferably with tar-

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taric or citric acid) in the proportion of from 5 to 10 parts of acid to 1000 parts of water. The liquor charged with the flavouring principles of the leaf may be preserved after any one of Pasteur's sterilizing methods, or it may be concentrated *in vacuo*, and thus brought to the degree of consistency which the extract is required to obtain.

The preserved liquor or extract will be serviceable for the same purposes as the vine leaf itself, provided care be taken, in the case of vintages to employ it in a proportion corresponding to the proportion of fresh leaves which will have to be used for the vintage. This liquor charged with the aromatic principles of the leaf or this extract from leaves contains most of the elements of a vine extract proper may be used for the purpose of increasing the proportion of dry extract of weak wines which, in years unpropitious to wine growers, will be very much appreciated by them, as an adjunct to either the vintage (grapes in process of wine production) or to ready made wine; the proportion in which it is added varying according to the result it is desired to attain.

I should like to call particular attention to the fact that these extract from leaves may be advantageously utilized in the manufacture of brandies of the Cognac type also for improving whiskies and for the fermentation of various beverages particularly those known as British wines.

I also may apply the method herein described to all other plants capable of being used in the preparation of drinks or foods.

Dated this 25th day of February 1899.

HASELTINE, LAKE & Co.,
45, Southampton Buildings, London, W.C.,
Agents for the Applicant.

COMPLETE SPECIFICATION.

Improvements relating to the Treatment of Leaves, Stems, and other Parts of Plants for Extracting and Utilizing the Aromatic Principles of the same, especially in the Manufacture of Alcoholic Liquors.

I, GEORGE EUGÈNE JACQUEMIN, of Malzéville, near Nancy, in the Republic of France, Agricultural Chemist, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The leaves of plants at various epochs of vegetable life are the seat of formation of certain principles which the plant utilizes for the benefit of some of its other organs, either immediately, or after having kept them in reserve for a time with the object of yielding them up at the opportune moment. To instance one fact, fruit is apt to acquire a characteristic flavour at the time of maturation, owing, in all probability, to the advent of this principle, the odour characterizing which is noticeable in the black currant leaf, for example; or the aromatic principle may have formed and become split up into two distinct principles, as in the case of apple-trees, pear-trees and the like where the odour or flavour of the fruit is not noticeable in the leaf beforehand.

The leaves of many plants, the fruits of which are clearly distinguishable by a flavour of their own, exhibit nothing whereby the prime cause of this well-defined flavour can be so much as surmised. Neither by crumpling or rolling them between the fingers, nor by pounding them in a porcelain mortar does the slightest odour or flavour of an aromatic or fragrant character become perceptible; and even by boiling them in water the flavour will generally not be rendered any more noticeable.

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It has occurred to me that these principles peculiar to certain leaves with regard to which the nature or operation which I am inclined to attribute to them,—as co-existent with the process of maturation of the fruit,—is not at all revealed in the leaf, might be likened to glucosides, as there is reason to suppose
 5 that bodies of this description which make their appearance in the fruit at a certain period of life of the plant, are confronted therein by a diastase, under the influence of which they become split up into glucose or saccharine matter and into an aromatic principle which imparts the peculiar character to the distinguishing flavour of the fruit.

10 Having explained this much by way of introduction, the object of the invention herein described is a practical application of the principles stated to the requirements of the manufacturing industry.

This I propose to effect by the following process.

I immerse leaves, say, of an apple tree or pear tree in a saccharine liquid
 15 of such density as to enable fermentation to commence and to terminate normally. It has been proved that leaven or yeast previously taken from a particular vintage, engenders in the wort it causes to ferment, a bouquet corresponding to the vintage from which it was taken, I therefore add some leaven or saccharomyces so selected as to ensure fermentation but not to bring out the fragrant
 20 or bouquet of the fruit. As soon as fermentation has set in there becomes very clearly noticeable an odour of apples or pears according to the kind of leaves treated, and by distilling these a species of brandy will be obtained, characterised by a fine flavour of the corresponding fruit.

This result clearly shows that the leaven, by means of a diastase, which it no
 25 doubt exudes, effects the division of this glucoside *i.e.* this principle peculiar to the leaves, into a special aromatic product and into sugar which will ferment together with the sugar in the liquid acting as a medium in this cellular life. I wish it understood however that my invention includes the process herein described no matter what theoretical explanation may be offered to account for
 30 the practical results which it enables me to attain.

A fermentation of the same kind obtained with vine leaves will yield a liquor of a vinous odour and, by distillation, a finely flavoured brandy. It will be understood that its flavour will be more delicate, the better the quality or nature of the stock from which the leaves are taken, or the better the wine which would
 35 in the ordinary course have been obtained from such stock.

In all these fermentations of the leaves of diverse fruit bearing plants, I have found that the odour of the aromatic principle extracted from the liquor was more marked, or intense, when distillation was proceeded with before fermentation was quite completed. On the other hand, as these aromatic prin-
 40 ciples are very volatile, a great deal of them is evolved during the fermentation process. Now it is important that the greatest possible quantity of this aroma should be intercepted, and to this end it will be necessary to pass the gases produced by fermentation through a condenser charged with alcohol, ether, spirits of petroleum, sulphuret of carbon, chloroform or the like which will dissolve
 45 the aroma thus given off; and the odorous gases should be conducted through a suitable apparatus with a view to separating the essential oils and intercepting even the faintest perfume which they carry with them.

It must further be understood that my invention comprises the application of the treatment just described not to leaves only but to all green portions of the
 50 plant, such as stalks, shoots, sprouts, the tendrils of vine foliage or the like.

Moreover, the method of operation just described is capable of undergoing a modification which I will now indicate.

Instead of submitting the leaves charged with the principles in question to fermentation, the aromatic principles may first be extracted from the leaves by
 55 decoction, infusion, digestion or any other treatment, with water, sacchariferous must, or any other suitable liquor, and then to induce fermentation in the presence of the aromatic principles thus extracted and separated from the leaves.

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This variation of my process involves no alteration in the general spirit in which it was conceived, seeing that the alcoholic fermentation which takes place, in any case affects only the particular principles contained in the leaves. The operation, therefore, takes the same course irrespective of whether the aromatic principles are still contained in the leaves or have been first separated therefrom. 5

I shall now state how I prefer to proceed in practice, taking the treatment of vine leaves as an instance.

The leaves of the foliage including stalks tendrils and the like are treated with boiling water or boiling saccharine must, wherein they are left to infuse for a number of hours. The must is separated from the leaves either before or after the complete cooling of the liquor, and having been brought to such a degree of richness in saccharine matter as will enable fermentation to commence and terminate in a normal manner, the leaven selected for the purpose is added to it, and it is then allowed to ferment. 10

A good result will also be obtained by boiling the leaves in the liquors above named, with or without subsequent infusion, or simply by causing the leaves to infuse and digest in a suitable liquid at a temperature below 100° Centigrade. The effect of these modifications will be simply to vary the degree of delicacy of the finer product. 15

Also, the quantity of vine leaves to be employed for every hectolitre of must to be fermented, will vary according to the result it is desired to obtain. It would seem that a proportion of from 2 to 5 kilogrammes of leaves per hectolitre should be used where it is proposed subsequently to distil the fermented liquor with a view to obtaining brandy. I may mention that by the expression "saccharine (or sacchariferous) must" I mean must containing sugar no matter of what origin or from what source, such as beet-sugar, glucose or the like. 20 25

All that has been stated above with regard to vine leaves equally applies to the leaves of apple, pear, raspberry, apricot, strawberry, cherry, plum, peach, rose and other plants, or trees. 30

Instead of fresh leaves, leaves which have been preserved by proper methods and brought from distant places, may be similarly treated.

Of the methods of preservation of leaves, the following appear to merit the preference:

The leaves are heaped in a cask, barrel or similar vessel, and a suitable proportion,—say about 5 or 10 *per cent.*, of leaven or yeast, or a liquid, in full fermentation, is poured over them. 35

Upon the heap of leaves there is further poured a syrup of sugar or glucose of as high a degree of concentration as possible; or glycerine may be used if desired; all the leaves being thoroughly flushed by, or submerged in, the liquor. The leaves thus treated will, for a long time, keep in a serviceable condition in a close vessel; while preservation may still further be assisted by an addition of an organic acid or an antiseptic. 40

Whatever the mode of preservation adopted, the leaves preserved will have to be used or treated after the same method that has been described with reference to fresh leaves. 45

My process also enables vine leaves, either from the same vine, or from a superior stock, to be used for the purpose of improving ordinary grape juice wines. The method of operation I adopt in this case being as follows.

The vine leaf in which are present,—either in the condition of glucosides or in any other forms,—those aromatic principles which, by migrating into the grape will presently contribute an important share to the development of the bouquet of the wine, may be employed either directly or indirectly for the amelioration of wines. 50

I have found that by means of vine leaves from a stock of recognised excellence, added to a somewhat poor vintage, the vinosity or bouquet of the wine 55

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from such vintage may be enhanced, by either leaving the wine to ferment spontaneously or by inducing its fermentation by means of leaven carefully selected from good stock. In the latter case the evolution produced by the leaven will be much more effective than it would be in ordinary wine juice left alone, because the leaf taken from a good stock brings with it, and imparts to the must, certain nutritive elements calculated to assist the evolution of the leaven selected, in a marked degree.

There are two ways of turning these observations to account, one consisting in adding from 100 to 500 grammes, or more, of vine leaves, per hectolitre of vintage to be treated; such leaves being either of the same or a superior stock, and having simply been washed first with acidulated water, and then with pure water, so as to get rid of any dust or foreign elements that may be adhering to them. The leaves will prove more effective if they are first chopped very finely by means of any suitable instrument, or if converted into a pulp before mixing with the grapes of the vintage. Leaves which have been preserved, no matter by what method, will have the same effect.

Or, preferably, infusions, decoctions or extracts of leaves, prepared beforehand in the following manner, may be used. The leaves after having been thoroughly cleansed, as above described, or having undergone any other convenient cleaning or purifying process, are chopped, pounded or otherwise divided,—or they may be left undivided,—are placed in an apparatus not unlike what is known as a digester, or in any other apparatus permitting their active principles to be extracted by means of cold or hot water acidulated (preferably with tartaric or citric acid) in the proportion of from 5 to 10 parts of acid to 1000 parts of water. The liquor charged with the flavouring principles of the leaf may be preserved after any one of Pasteur's sterilizing methods, or it may be concentrated *in vacuo*, and thus brought to the degree of consistency which the extract is required to obtain.

The preserved liquor or extract will be serviceable for the same purposes as the vine leaf itself provided care be taken, in the case of vintages to employ it in a proportion corresponding to the proportion of fresh leaves which would have been used for the vintage. This liquor charged with the aromatic principles of the leaf, or this extract from leaves, contains most of the elements of a wine extract proper and may be used for the purpose of increasing the proportion of dry extract in weak wines, which, in years unpropitious to wine growers, will be very much appreciated by them, as an adjunct to either the vintage (grapes in process of wine production) or to ready made wine; the proportion in which it is added varying according to the result it is desired to attain.

I should like to call particular attention to the fact that these extracts from leaves may be advantageously utilized in the manufacture of brandies of the Cognac type also for improving whiskies and for the fermentation of various beverages particularly those known as British wines.

It should be remarked that the leaves are not the only parts of the plant adapted for use under the conditions before explained; the result of my experiments proving particularly with respect to the vine, that the products of glucosides normally engendered in the leaves and in all the green parts of the vine, and communicated to the grapes in which it produces part of the flavour at the period of maturity, exists also in the stem of the vine. The quantity of glucosides that are found in the stem after the vintage, is quite notable and probably constitutes a residue of the glucosides engendered in the leaves on its passage into the fruit.

I may here remark that also the stripped stalks of the grape can also equally well be employed. This green part, supporting the separate grapes serving for the passage of the glucosides engendered in the leaves, to the fruit, contains at all times, even after the maturity of the grape, a notable proportion of glucosides which may be easily extracted by any convenient process, digestion, infusion or the like.

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I have noticed that the stalks, which are not green, taken from the fermentation of the wine or remaining after the stripping-off of the grapes before wine-making, and also generally the whole of the skins and residues either before or after fermentation even after long preservation after being separated from the wine, still contain glucosides which may be extracted by any of the methods before indicated. This is easily explained, as the stalks have not been completely exhausted by their immersion in the new wine, and furthermore the skins of the grapes which remain in the residues, contain a large quantity of these glucosides which have been engendered in the leaves, and then passed into the fruit. The infusion of the skins as effected in ordinary wine-making cannot extract the whole of the principles contained in the tissues of the skins; it therefore follows that a notable portion of these principles may be recovered by a suitable treatment of the residues.

Lastly the stones of the grapes also contain a certain quantity of glucosides, derived from the leaves, and existing after the finish of the ordinary wine-making process, owing to the principles being protected by the solid shell or husk during the fermentation of the wine: and they may be easily extracted after grinding or pounding either separately or mixed with the other residues for simultaneous treatment.

I also may apply the method herein described to all other plants capable of being used in the preparation of beverages or the like.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. The process for the production of the aromatic principles hereinbefore described by fermentation of the leaves or other green parts of plants such as the skins, stalks, stones, branches or stalks of grape-bunches, and stems, whether fresh or preserved, such process being applicable specially to the vine but also applicable to apple, pear, raspberry, apricot, strawberry, cherry, plum, peach, rose and other plants, and the aromatic principles being extracted, as may be desired, either by distillation or by means of agents such as ether, spirits of petroleum, sulphuret of carbon, chloroform or the like, or by any other means as hereinbefore described and specified.

2. The modification of the process for the production of aromatic principles as claimed in Claim 1, consisting in making an extract from the leaves or other parts of plants by decoction, infusion, digestion or the like in water, in preparing a sweet wort or other convenient liquor for causing fermentation in the said extract, after having previously separated-out the leaves and other parts of the plant, substantially as hereinbefore described and specified.

3. A process for ameliorating wine, obtained by ordinary methods, in mixing therewith, at the vintage, the leaves and other green parts of the vine, either wholly or in a divided state and either fresh or in a preserved condition, or mixing therewith liquids, either fresh or preserved, which contain in solution the active principles of the leaves: or extracts, either fresh or preserved, of such, substantially as hereinbefore described and specified.

4. The utilization of leaves or other parts of vine or other plants or liquids or extracts containing the principles of the leaves or other parts of the plant, with the object of furnishing this extract to wines or beverages requiring the same, as well as for other purposes hereinbefore mentioned, substantially as specified.

5. The application of the process of fermentation as claimed in Claim 1, for producing beverages such as wine, brandy, beverages known as English wines, or for ameliorating such beverages, wines, brandies, whiskeys and the like, by distillation of the fermented liquids obtained in accordance with Claim 1, or by adding to such beverages aromatic principles under any form either direct or indirectly, substantially as hereinbefore described and specified.

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6. The various modes of operating as herein described for obtaining the fermented liquids charged with aromatic principles, or the extracts of these aromatic principles, or special liquids charged with such extracts, for the production of beverages and for ameliorating such beverages, substantially as
5 hereinbefore described and specified.

Dated this 21st day of November 1899.

HASELTINE, LAKE & Co.,
45, Southampton Buildings, London, W.C.,
Agents for the Applicant.

Redhill: Printed for Her Majesty's Stationery Office, by Malcomson & Co., Ltd.—1900.

*Heels leaves of apples
is for obtaining fermented
principles & beverages*